Effect of high pressure treatment on thermal and rheological properties of chickpea (*Cicer arietinum* L.) flour dispersions and pastes

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Introduction

Chickpea is a crop of economic importance and also an important source of protein in the diet of people in numerous countries [1]. Dispersions of chickpea flours are used in the production of several convenience foods including fried snacks and sweets [2], as well as in emulsified meat products [3]. It is still desirable to develop chickpea based products or partially substitute wheat flour by chickpea flour in different food products. High hydrostatic pressure (HHP) provides the possibility to produce foods with novel textures [4]. To achieve the desired product functionality and texture, the understanding of pressure-induced gelatinization of starch is vital for applications of HPP treatment in starch-containing products. Rheology and differential scanning calorimetry (DSC) provide information at physical and macroscopic level indicating changes associated with gelatinization or denaturation processes [5]. The objective of this work was to evaluate the effect of HHP treatment on rheological and thermal properties of chickpea flour slurry. HHP treated dispersions were studied as function of pressure level (0, 150, 300, 450 and 600 MPa), slurry concentration (with 1:5, 1:4, 1:3 and 1:2 flour-to-water ratios) and temperature. For that, flour-water suspensions were pre-treated with pressure and subsequently analyzed for changes in their properties by an isothermal heating process carried out at 75 °C for 15 min and at 90 °C for 5 min.

Experimental Methods

Materials

Spanish chickpea (cv. Castellano) flour used was a commercially available product donated by the flour miller industry *Los Pisones* (Zamora, Spain). Chickpea flour was supplied packed in polyethylene pouches (500 g) and stored in watertight cabins (10 °C and 73 ± 3% relative humidity) until use.

Sample preparation

Rice flour slurries were prepared at four concentrations yielding 1:5, 1:4, 1:3 and 1:2 flour-to-water ratios. At each case, the measured quantity of water was added in small portions and the flour was initially stirred using a glass rod, to break the lumps and to form a smooth suspension [6]. Next, the sample was kept for half-an-hour at room temperature for hydration, stirring constantly at 900 rpm before subjecting it to rheological or thermal measurements. Slurries (200 mL) were vacuum packaged in flexible bags type Doypack® (Polyskin XL, Amcor

Flexiblehispania, Granollers, Spain), introduced into the pressure unit filled with pressure medium (water) and then treated by high-pressure (150, 300, 450, 600 MPa/25 °C/15 min). HHP treatments were performed using a Stansted Fluid Power Iso-lab 900 High Pressure Food Processor (Model: FPG7100:9/2C, Stansted Fluid Power Ltd., Harlow, Essex, UK), with 2000 mL capacity, maximum pressure of 600 MPa, and maximum temperature of 60 °C.

Dynamic rheological measurements

A Bohlin CVR 50 controlled stress rheometer (Bohlin Instruments Ltd., Cirencester, UK) was used to conduct rheological measurements in combination with a four-bladed cruciform vane geometry (diameter = 25 mm and height = 40 mm), which rotates inside a 27-mm-diameter serrated cup with 0.5 mm deep serrations, and a solvent trap to minimize moisture loss during tests. SAOS isothermal measurements were carried out at three selected temperatures (25, 75 and 90 °C). Sample temperature was internally controlled via a computer using a Bohlin Rheology fluid circulating bath KTB-30 (also from Bohlin Instruments Ltd.). In order to induce a paste, flour dispersions were isothermally heated in the vane geometry to 75 and 90 °C by using the pre-condition option for 15 and 5 min, respectively with controlled stress at 0 Pa before the actual measurements. Frequency sweep oscillatory tests were performed at variable frequencies over the range 0.1-100 rad s^{-1}, keeping the amplitude stress at a constant value within the LVE region.

Thermal properties

A DSC (TA Q1000, TA Instruments, New Castle, DE, USA) was employed and calibrated with indium and sapphire for temperature and heat capacity values. Slurry samples, weighing around 15 mg (±0.002) were capsulated in hermetically sealed aluminum volatile pans. Thermal scans were performed from 25 to 100 °C at a heating rate of 10 °C min^{-1}. An empty pan was used as a reference and dry nitrogen at a flow rate of 50 mL min^{-1}, was used as the purge gas. Thermal transitions were measured in terms of onset (T_0), peak (T_p) and conclusion (T_c) gelatinization temperatures. The gelatinization temperature range (R) was computed as (T_c - T_0). The enthalpy (ΔH_{gel}) of the transition was calculated from the area of the peak endotherm. The peak height index (PHI) was calculated by the ratio $\Delta H_{gel}/(T_p - T_0)$, as described by Kaur and Singh [1].

Results and Discussion

Rheological properties

In order to investigate the HHP effect on the flour slurries, samples were analyzed directly after treatment. The viscoelastic behavior of the chickpea flour slurries with 1:2 flour-to-water ratio at 25 °C is illustrated in Fig. 1. The behavior of unpressurized and pressurized dispersions at 25 °C resembled that of an entangled system, with G” > G’ until the crossover frequency was reached. Slurries underwent significant increases in both moduli with flour concentration and applied pressure. Following increasing trends of both G’ and G”, there was a systematic increase in η* with applied pressure treatment indicative of the presence of higher entanglement density, demonstrating that the treatment of chickpea slurries with increasing pressure causes increasing gelatinization of starch. Pressure-induced melting of sorghum starch granules started at pressures > 300 MPa and complete gelatinization was obtained after treatment with 600 MPa [4]. It was also clear that the swelling of starch granules was correlated with the increase in η_{initial} [7].

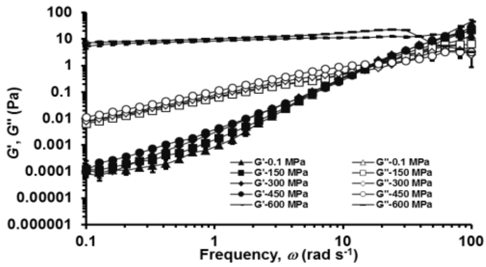

Figure 1. Effect of HHP treatment on mechanical spectra of 1:2 chickpea flour slurry at 25 °C

A dispersion can be converted into a paste under various conditions, such as temperature change. Mechanical spectra of heat-induced pastes at 75 °C for 15 min (for samples of 1:5 and 1:3 flour-water slurries) before and after HPP treatments are presented in Figs. 2 and 3. Similar results were obtained for heat-induced pastes at 90 °C for 5 min (not shown).

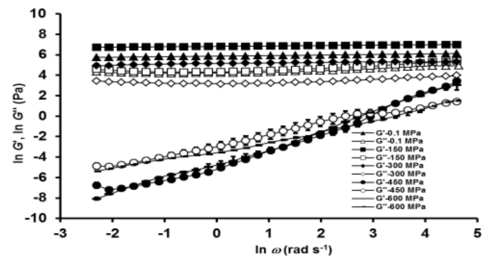

Figure 2. Effect of HHP treatment on mechanical spectra of 1:5 chickpea flour paste induced at 75 °C for 15 min

Elasticity (*G*') of thermally induced flour paste increased as function of chickpea flour concentration and decreased with increasing applied pressure in proportion with the extent of high-pressure-induced gelatinization of starch. It is well known that gelatinization is an irreversible melting phase transition [4]. Consequently, in the subsequent heating process weaker pastes are formed with increasing the amount of pre-gelatinized starch and after only melting of the remaining crystallites. The effect of HHP treatment was also more pronounced at the higher water contents. Even due to earlier pressure-induced gelatinization, weak gel behavior was not observed for pressure treated (600 MPa) samples of 1:5 flour-water slurry (Fig. 2). Certainly, frequency sweeps provides information on physical changes that can indicate the degree of gelatinization.

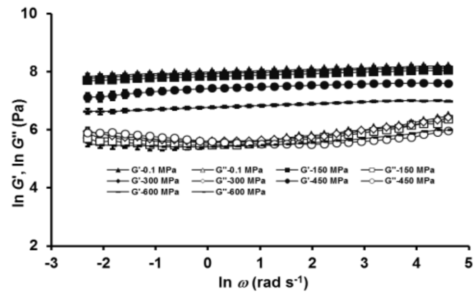

Figure 3. Effect of HHP treatment on mechanical spectra of 1:3 chickpea flour paste induced at 75 °C for 15 min

Thermal properties

The melting enthalpies of the pressure-treated slurries showed progressive gelatinization of starch as either concentration or pressure levels increased. The enthalpy value increased as concentration increased. It was also found that the enthalpy decreased with increasing pressure level. Conversely, chickpea flour dispersions subjected to 600 MPa for 15 min showed no peak and hence no enthalpy value, suggesting complete HHP-induced gelatinization of starch. Stolt et al. [8] found a complete loss of birefringence in barley starch after treatment at 600 MPa for 15 min.

Table 1. Effects of HHP treatment and concentration on thermal properties of chickpea flour slurry

HPP/Ratio	ΔH_{gel} (J/g)	PHI
0.1 MPa/1:5	1.14 ± 0.01^{aC}	$0.15\pm0.00^{a,bB,C}$
150 MPa/1:5	1.41 ± 0.27^{aC}	0.17 ± 0.05^{aC}
300 MPa/1:5	1.12 ± 0.01^{aD}	$0.10\pm0.00^{a,bD}$
450 MPa/1:5	1.15 ± 0.01^{aC}	0.08 ± 0.00^{bC}
600 MPa/1:5	ND	-
0.1 MPa/1:4	1.32 ± 0.06^{cC}	0.13 ± 0.01^{cC}
150 MPa/1:4	3.24 ± 0.03^{aB}	0.54 ± 0.00^{aB}
300 MPa/1:4	1.70 ± 0.01^{bC}	0.18 ± 0.00^{bC}
450 MPa/1:4	0.71 ± 0.01^{dD}	0.07 ± 0.00^{dC}
600 MPa/1:4	ND	-
0.1 MPa/1:3	1.79 ± 0.16^{cB}	0.19 ± 0.03^{cB}
150 MPa/1:3	3.32 ± 0.02^{aB}	0.47 ± 0.00^{aB}
300 MPa/1:3	2.48 ± 0.02^{bB}	0.37 ± 0.00^{bB}
450 MPa/1:3	1.93 ± 0.02^{cB}	0.19 ± 0.00^{cB}
600 MPa/1:3	ND	-
0.1 MPa/1:2	2.50 ± 0.09^{dA}	0.29 ± 0.01^{cA}
150 MPa/1:2	4.21 ± 0.03^{aA}	0.64 ± 0.01^{aA}
300 MPa/1:2	3.19 ± 0.03^{bA}	0.40 ± 0.00^{bA}
450 MPa/1:2	2.79 ± 0.02^{cA}	0.37 ± 0.00^{bA}
600 MPa/1:2	ND	-

$^{a\text{-}d}$For the same concentration, parameter means without the same letter are significantly different (P < 0.01). $^{A\text{-}D}$For the same HHP treatment, parameter means without the same letter are significantly different (P < 0.01).

Concluding Remarks

Chickpea flour dispersions were partial or totally gelatinized under pressure but the quality of the yielded pastes was falling with increasing pressure level. It may be hypothesized that the addition of HHP-treated chickpea slurries (600 MPa, 15 min) in unpressurized similar batter based products can offer easier flow characteristics during heating, preparation and handling. On the other hand, data presented provide a useful fingerprint of rheological behavior, which plays an important role in optimizing sensory quality of final food products.

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